

Refine Search

Search Results -

Terms	Documents
L9 and L2	5

US Pre-Grant Publication Full-Text Database
US Patents Full-Text Database
US OCR Full-Text Database
EPO Abstracts Database
JPO Abstracts Database
Derwent World Patents Index
IBM Technical Disclosure Bulletins

Database: SCOPUS All Text Database
EPO Abstracts Database
JPO Abstracts Database
Derwent World Patents Index
IBM Technical Disclosure Bulletins

Search:

L10

Refine Search

Recall Tex

Clear

Interrupt

Search History

DATE: Thursday, September 02, 2004 [Printable Copy](#) [Create Case](#)

Set
Name Query
side by
side

Hit Set
Count Name
result set

DB=PGPB,USPT,USOC,EPAB,JPAB,DWPI,TDBD; PLUR=YES; OP=ADJ

<u>L10</u>	L9 and l2	5	<u>L10</u>
<u>L9</u>	L8 with (water or H\$1O)	622	<u>L9</u>
<u>L8</u>	L5 with ag with v with o	8619	<u>L8</u>
<u>L7</u>	L5 same ag same v same o	81481	<u>L7</u>
<u>L6</u>	L5 and ag and v and o	220837	<u>L6</u>
<u>L5</u>	(li or an or k or rb or cs or ti or mg or ca or sr or ba or cu or zn or cd or pb or cr or au or al or fe or co or ni or mo)	23635849	<u>L5</u>
<u>L4</u>	L3 and ag and v and o	540	<u>L4</u>
<u>L3</u>	l1 and (li or an or k or rb or cs or ti or mg or ca or sr or ba or cu or zn or cd or pb or cr or au or al or fe or co or ni or mo)	3306	<u>L3</u>
<u>L2</u>	L1 and (502/\$ or 429/\$ or 423/\$ or 502/\$ or 549/\$)	1522	<u>L2</u>
<u>L1</u>	silver and vanadium and metal oxide and catalyst	3312	<u>L1</u>

Hit List

Clear	Generate Collection	Print	Fwd Refs	Bkwd Refs
Generate OACs				

Search Results - Record(s) 1 through 5 of 5 returned.

1. Document ID: US 20040116737 A1

Using default format because multiple data bases are involved.

L10: Entry 1 of 5

File: PGPB

Jun 17, 2004

PGPUB-DOCUMENT-NUMBER: 20040116737

PGPUB-FILING-TYPE: new

DOCUMENT-IDENTIFIER: US 20040116737 A1

TITLE: Nox treated mixed metal oxide catalyst

PUBLICATION-DATE: June 17, 2004

INVENTOR-INFORMATION:

NAME	CITY	STATE	COUNTRY	RULE-47
Gaffney, Anne Mae	West Chester	PA	US	
Heffner, Michele Doreen	Chalfont	PA	US	
Song, Ruozhi	Wilmington	DE	US	

US-CL-CURRENT: 562/546; 502/302, 562/547

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KMTC	Drawn D
------	-------	----------	-------	--------	----------------	------	-----------	-----------	-------------	--------	------	---------

2. Document ID: US 5881358 A

L10: Entry 2 of 5

File: USPT

Mar 9, 1999

US-PAT-NO: 5881358

DOCUMENT-IDENTIFIER: US 5881358 A

TITLE: Composition for extracting transition metal and method for extracting transition metal using the same

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KMTC	Drawn D
------	-------	----------	-------	--------	----------------	------	-----------	-----------	-------------	--------	------	---------

3. Document ID: US 4272409 A

L10: Entry 3 of 5

File: USPT

Jun 9, 1981

US-PAT-NO: 4272409

DOCUMENT-IDENTIFIER: US 4272409 A

** See image for Certificate of Correction **

TITLE: Aluminosilicate sols and powders

[Full](#) | [Title](#) | [Citation](#) | [Front](#) | [Review](#) | [Classification](#) | [Date](#) | [Reference](#) | [Search](#) | [Print](#) | [Fwd Refs](#) | [Bkwd Refs](#) | [Claims](#) | [KMC](#) | [Drawn D](#)

4. Document ID: US 4257874 A

L10: Entry 4 of 5

File: USPT

Mar 24, 1981

US-PAT-NO: 4257874

DOCUMENT-IDENTIFIER: US 4257874 A

**** See image for Certificate of Correction ****

TITLE: Petroleum refinery processes using catalyst of aluminosilicate sols and powders

[Full](#) | [Title](#) | [Citation](#) | [Front](#) | [Review](#) | [Classification](#) | [Date](#) | [Reference](#) | [Search](#) | [Print](#) | [Fwd Refs](#) | [Bkwd Refs](#) | [Claims](#) | [KMC](#) | [Drawn D](#)

5. Document ID: US 4217240 A

L10: Entry 5 of 5

File: USPT

Aug 12, 1980

US-PAT-NO: 4217240

DOCUMENT-IDENTIFIER: US 4217240 A

TITLE: Stable aluminosilicate aquasols having uniform size particles and their preparation

[Full](#) | [Title](#) | [Citation](#) | [Front](#) | [Review](#) | [Classification](#) | [Date](#) | [Reference](#) | [Search](#) | [Print](#) | [Fwd Refs](#) | [Bkwd Refs](#) | [Claims](#) | [KMC](#) | [Drawn D](#)

[Clear](#) [Generate Collection](#) [Print](#) [Fwd Refs](#) [Bkwd Refs](#) [Generate OACS](#)

Terms	Documents
L9 and L2	5

Display Format: [-] [Change Format](#)

[Previous Page](#)

[Next Page](#)

[Go to Doc#](#)

d his

(FILE 'HOME' ENTERED AT 17:26:00 ON 02 SEP 2004)

FILE 'CAPLUS' ENTERED AT 17:28:02 ON 02 SEP 2004

L1 55 S MULTIMETAL OXIDE
L2 39 S MULTI-METAL OXIDE
L3 92 S L1 OR L2
L4 60239 S METAL OXIDE
L5 60280 S L4 OR L3
L6 83 S L5 AND AG AND V AND O
L7 6200095 S LI OR MA OR K OR RB OR CS OR TL OR MG OR CA OR SR OR BA OR CU
L8 75 S L7 AND L6
L9 14 S L8 AND (WATER OR H2O)
L10 9 S L9 AND PY<1998

=>

=> d ibib abs hitstr

L10 ANSWER 1 OF 9 CAPLUS COPYRIGHT 2004 ACS on STN
ACCESSION NUMBER: 1997:381724 CAPLUS
DOCUMENT NUMBER: 127:141945
TITLE: Electrochemistry of the copper-nickel series of heteropolymetallic complexes $[(\mu_4-\text{O})\{\text{NC}_5\text{H}_4(\text{C}(\text{O})\text{NEt}_2)_3\}_4\text{Cu}_{4-x}\{\text{Ni}(\text{H}_2\text{O})\}_x\text{Cl}_6]$ ($x = 0-4$)
AUTHOR(S): Workie, Bizuneh; Dube, Christopher E.; Aksu, Levent; Kounaves, Samuel P.; Robbat, Albert, Jr.; Davies, Geoffrey
CORPORATE SOURCE: Dep. of Chem., Tufts Univ., Medford, MA, 02155, USA
SOURCE: Journal of the Chemical Society, Dalton Transactions: Inorganic Chemistry (1997), (10), 1739-1745
CODEN: JCDTBI; ISSN: 0300-9246
PUBLISHER: Royal Society of Chemistry
DOCUMENT TYPE: Journal
LANGUAGE: English
AB The electrochem. of the tetrานuclear copper-nickel heteropolymetallic complexes $[(\mu_4-\text{O})\text{L}_4\text{Cu}_{4-x}\{\text{Ni}(\text{H}_2\text{O})\}_x\text{Cl}_6]$ [$x = 0-4$, L = N,N-diethylnicotinamide (denc)] were studied at a platinum electrode in DMSO with 0.20M tetrabutylammonium hexafluorophosphate as supporting electrolyte. At potentials more cathodic than -1.0 V the complexes are electrodeposited as Cu-Ni alloy and metal oxide films and display a complicated set of cyclic voltammographs. The voltammographs of all the Cu-containing complexes show a quasi-reversible redox couple in the potential range 0.250 to -0.450 V vs. Ag-AgPF₆ (0.01M)-CH₃CN. As the number of Cu atoms decreases in the complex, the peak currents i_{pa} and i_{pc} decrease proportionally and the peak potential shifts anodically. The cyclovoltammetric (CV) results indicate that electron transfer initially occurs only to the CuII centers and that the electron-transfer reaction appears to be quasi-reversible. Using steady-state voltammetry at an ultramicroelectrode in combination with chronoamperometry at a microelectrode and exhaustive electrolysis at a Hg-pool electrode, the number of electrons (n) transferred for this initial reduction of the Cu₄, Cu₃Ni, Cu₂Ni₂ and CuNi₃ complexes were 3.1, 2.1, 1.8 and 0.57, resp. The diffusion coefficient for all the complexes was $2.2(+0.1) \times 10^{-6}$ cm² s⁻¹. The electronic spectrum of the Cu₄ complex taken after exhaustive electrolysis shows that one quarter of the Cu atoms remain in the CuII form and that the CuI complex remains stable. Since only a single CV peak results for all of the complexes, the electron transfer is most likely consecutive with very closely spaced E° potentials. A model based on statistically determined electron transfer to CuII in particular faces is also proposed.
REFERENCE COUNT: 31 THERE ARE 31 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

=> d 2-9 ibib abs hitstr

L10 ANSWER 2 OF 9 CAPLUS COPYRIGHT 2004 ACS on STN
ACCESSION NUMBER: 1993:580403 CAPLUS
DOCUMENT NUMBER: 119:180403
TITLE: Preparation of α,β -unsatd. nitriles by catalytic vapor-phase ammonoxidation of saturated hydrocarbons
INVENTOR(S): Blanchard, Gilbert; Bordes, Elisabeth; Ferre, Gilbert
PATENT ASSIGNEE(S): Rhone-Poulenc Chimie SA, Fr.
SOURCE: Eur. Pat. Appl., 16 pp.
CODEN: EPXXDW
DOCUMENT TYPE: Patent
LANGUAGE: French
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
------------	------	------	-----------------	------

EP 547981	A1	19930623	EP 1992-420446	19921203 <--
EP 547981	B1	19950809		
R: BE, DE, ES, FR, GB, IT, NL, PT				
FR 2684989	A1	19930618	FR 1991-15843	19911216 <--
FR 2684989	B1	19940204		
ES 2076727	T3	19951101	ES 1992-420446	19921203 <--
JP 06135921	A2	19940517	JP 1992-351423	19921208 <--
JP 06092355	B4	19941116		
CA 2085451	AA	19930617	CA 1992-2085451	19921215 <--
CA 2085451	C	19970121		
CN 1075314	A	19930818	CN 1992-114404	19921215 <--
CN 1032647	B	19960828		
RU 2060247	C1	19960520	RU 1992-4546	19921215 <--
US 5334743	A	19940802	US 1992-991170	19921216 <--
			FR 1991-15843	19911216

PRIORITY APPLN. INFO.:

CASREACT 119:180403

AB Alkanes are converted to α, β -unsatd. nitriles by ammoxidn.. in the vapor phase in the presence of solid catalysts, the active phase of which consists of Mo, V, and O, along with at least one of the elements Mn, Zn, Co, Cu, Li, Na, K, or Ag. The process is particularly useful for the ammoxidn.. of propane to give a mixture of acrylonitrile and propylene. Thus, catalysts such as Mn0.8V1.6Mo0.406, Zn0.85V1.7Mo0.306, or Co0.9V1.7Mo0.206 (preps. given) were used in the ammoxidn.. of propane (NH₃, O₂, H₂O vapor, He flow, 1.3 bar) at various temps. to afford acrylonitrile and propylene mixts.

L10 ANSWER 3 OF 9 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1991:186328 CAPLUS

DOCUMENT NUMBER: 114:186328

TITLE: Molybdenum- and phosphorus-based oxidation catalysts for the production of methacrylic acid from methacrolein, isobutyraldehyde, and isobutyric acid

INVENTOR(S): Kurimoto, Ikuo; Hashiba, Hideto; Onodera, Hideo; Aoki, Yukio

PATENT ASSIGNEE(S): Nippon Shokubai Kagaku Kogyo Co., Ltd., Japan

SOURCE: Eur. Pat. Appl., 32 pp.

CODEN: EPXXDW

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 415347	A2	19910306	EP 1990-116480	19900828 <--
EP 415347	A3	19920226		
EP 415347	B1	19941026		
EP 415347	B2	20020605		
R: BE, DE, ES, FR, GB, IT, NL				
JP 03086242	A2	19910411	JP 1989-220380	19890829 <--
JP 04090853	A2	19920324	JP 1990-206857	19900806 <--
JP 2994706	B2	19991227		
US 5153162	A	19921006	US 1990-574139	19900829 <--
			JP 1989-220380	A 19890829
			JP 1990-206857	A 19900806

PRIORITY APPLN. INFO.:

AB Catalysts for the title use contain an active layer of Mo_aPb_bAc_cCe_dBdDfO_x (I, A = As, Sb, Ge, Bi, Zr, Ce, or Se; B = Cu, Fe, Cr, Ni, Mn, Co, Sn, Ag, Zn, Pd, Rh, or Te; C = V, W, or Nb; D = alkali metal, alkaline-earth metal, or Tl; a, b, c, d, e, f, and x = atomic ratio for each atom; when a = 12, b = 0.5-4, c = 0-5, d = 0-3, e = 0-4, f = 0.01-4, x = number of oxidation states of all elements) and are prepared by mixing compds. of the elemental components of the oxide of I, heating the mixed compds. to prepare a slurry or a solution, spraying the slurry or solution onto an inert carrier, and calcining. Adding arsenious acid 123.7, 85% H₃PO₄ 523.8, and CsNO₃ 812.3 g in 4 L HNO₃ and 5 L H₂O to 8830 g NH₄ paramolybdate 8830 and 531.4 g NH₄ metavanadate, stirring, spraying the heated slurry onto a

silicon carbide carrier, and calcining at 400° for 3 h gave a catalyst with composition Mo₁₂P_{1.09}V_{1.09}Ce_{1.0}As_{0.30}x. A vapor mixture containing methacrolein 3.5, isobutylene 0.04, methacrylic acid (II) and acetic acid 0.24, steam 20, O₂ 9.0, and others 67.2 volume% was oxidized with this catalyst to give II with 87.1% selectivity and 89.2% conversion.

L10 ANSWER 4 OF 9 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1985:166979 CAPLUS

DOCUMENT NUMBER: 102:166979

TITLE: Vinylcyclohexenedicarboxaldehydes

PATENT ASSIGNEE(S): Nippon Zeon Co., Ltd., Japan

SOURCE: Jpn. Kokai Tokkyo Koho, 5 pp.

CODEN: JKXXAF

DOCUMENT TYPE: Patent

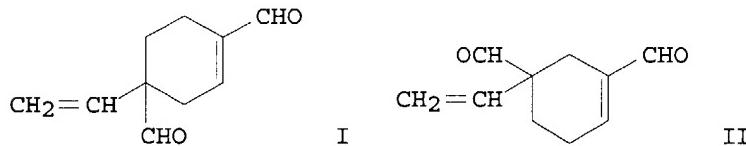
LANGUAGE: Japanese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 59204148	A2	19841119	JP 1983-75282	19830428 <--
PRIORITY APPLN. INFO.:			JP 1983-75282	19830428

GI



AB The title compds. (I and II) were prepared by oxidation of C5-compds. containing Me groups by mol. O in the presence of mixed oxides of (1) Sn, (2) Co, Ni, Se, Bi, P, U, Zr, Cu, Pb, Mn, W, V, and/or Cr, (3) groups I and II and/or Ti, (4) Al, In, Mb, Ta, Ga, Th, Pd, Ru, Rh, Ge, Ag, B, As, Se, Si, Ti, Mo, Te, Sb, and/or rare earth metals. Thus, a small quantity of concentrated HNO₃ was added to a mixture of 118.7 g Sn and 100 mL H₂O, the resulting mixture stirred, evaporated to dryness, dried at 120° for 8 h, calcined at 350° for 4 h, pulverized to < 100 mesh, 11.5 g 85% H₃PO₄ and 200 mL H₂O were added, the resulting mixture was dried at 120° for 8 h, calcined at 350° for 4 h, pulverized to < 100 mesh, adsorbed to about 30 wt % on a silicon carbide support, heated at 400° for 2 h, and then calcined at 900° for 6 h to give a Sn₁P_{0.1} catalyst, over which (50 mL) in a reactor heated at 470° was passed (SV 3500 h⁻¹) a 2:20:78 (molar ratio) mixture of tert-amyl alc., air, and steam to give a mixture containing 5.7% I and 94.3% II with 31% selectivity and 42% conversion.

L10 ANSWER 5 OF 9 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1984:571916 CAPLUS

DOCUMENT NUMBER: 101:171916

TITLE: Unsaturated acids and esters by oxidative condensation

INVENTOR(S): Grasselli, Robert Karl; Guttmann, Andrew Tytus

PATENT ASSIGNEE(S): Standard Oil Co., USA

SOURCE: Eur. Pat. Appl., 20 pp.

CODEN: EPXXDW

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 111605	A1	19840627	EP 1982-306820	19821221 <--
EP 111605	B1	19860409		

R: BE, CH, DE, FR, GB, IT, LI, LU, NL, SE

CA 1193613

A1 19850917

CA 1982-417257

19821208 <--

PRIORITY APPLN. INFO.:

EP 1982-306820

19821221

AB Oxidation catalysts for the selective oxidation of MeOAc [79-20-9] or Me propionate [554-12-1] with MeOH [67-56-1] (or Me₂O [115-10-6]) at 330° in the presence of (in N dilution) have the general formula AaBbCcPO_x, A is alkali metal, alkaline earth metal, or Tl; a = 0-1.5; B is Fe, Co, Ni, V, Cr, Mn, Cu, Pt, Pd, Rh, Ru, or Ag; (b = 0.01-3.0); C is Sc, Y, Ti, W, Nb, Si, La, Ge, Ce, Th, U, Mo, Bi, Sb, or Te; (c = 0-2.0); and x is the number required to satisfy valence requirements. Thus, 44.1 g Al₂O₃.xH₂O in 170 mL water at 70° was stirred with aqueous (NH₄)₂HPO₄, aqueous Th(NO₃)₄.4H₂O and Cu(NO₃)₂.3H₂O, and aqueous KNO₃, the slurry was evaporated at 90° and dried at 120-125°, and the dried material was heated 3 h at 290° and 3 h at 350°, ground, screened to 20-35 mesh, and calcined 5 h at 500°. A fixed-bed reactor containing the catalyst was fed at MeOAc-MeOH-O-N ratio 10:1:0.8:7.2 to give 74.9% Me acrylate [96-33-3] and 10.9% acrylic acid [79-10-7].

L10 ANSWER 6 OF 9 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1971:124849 CAPLUS

DOCUMENT NUMBER: 74:124849

TITLE: Acetic acid

INVENTOR(S): Nakajima, Kazuhisa; Sato, Tsuneo

PATENT ASSIGNEE(S): Japan Synthetic Chemical Industry Co., Ltd.

SOURCE: Jpn. Tokkyo Koho, 3 pp.

CODEN: JAXXAD

DOCUMENT TYPE: Patent

LANGUAGE: Japanese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 45041571	B4	19701226	JP	19671213 <--

AB Butene is oxidized at 220-350° with O-containing gases in the presence of steam over V_XO catalysts (X = Cr, Mn, Cu, Zn, Bi, Ag, Pb, Zr, and (or) Nb and V -X = 1:0.01-1.5). Thus NH₄VO₃ and Cr(AcO)₃.H₂O in hot H₂O is mixed with silica colloid aqueous solution, evaporated, heated to 480° during 8 hr in air, roasted 8 hr at 480°, and crushed to give a catalyst with V-Cr-O 1:0.1:2.8. Oxidation of butene-air-steam mixture over this gave 49.2% AcOH.

L10 ANSWER 7 OF 9 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1970:54769 CAPLUS

DOCUMENT NUMBER: 72:54769

TITLE: Catalysts for production of acrylic acid by vapor-phase oxidation of acrolein

PATENT ASSIGNEE(S): Toa Gosei Chemical Industry Co., Ltd.; Institute of Physical and Chemical Research

SOURCE: Brit., 8 pp.
CODEN: BRXXAA

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
GB 1170851		19691119		<--
DE 1643087			DE	
US 3567772		19710000	US	<--

PRIORITY APPLN. INFO.: JP 19660330

AB Catalyst complexes consisting of mixts. of Mo, V, and Al oxides and oxides of Cu, Ge, U, Mn, Ba, Si, Au, Se, Ca, Ti, Sr, Ag, Mg, B, Sn, W, Pb, or As are used for the vapor-phase oxidation of acrolein (I) to give high yields of high-purity acrylic acid (II). Thus,

92.7 g 6-10 mesh Al sponge was warmed with an aqueous solution containing NH₄ molybdate 22.45, NH₄VO₃ 2.36, and Cu(NO₃)₂ 5.52 g and the mixture was stirred and dried at 120° before being heated in air at a temperature increasing to 285°. The catalyst was screened and 50 ml was replaced in the reaction tube and heated to 300°. A feed stream containing 3.2% I, O at a mole ratio of 2.4, and H₂O at a mole ratio of 15 with respect to I, was introduced at 1230 hr⁻¹ space velocity and the temperature was increased to 400°. The oxidation was continued for 3 hr over a 100:10:3:10 MoO₃-V2O₅-Al₂O₃-CuO catalyst and a 99.9% conversion was obtained giving a 91.1% yield of 95.2% pure II. Conversions of 99.8 and 98.4% were obtained at 325 and 300°, resp., giving yields of 95.7 and 96% of 97.3 and 97.6% pure II. Other catalysts used contained GeO₂, UO₃, MnO₂, BaO, SiO₂, Au₂O₃, SeO₂, CoO, SrO, CuO mixed with GeO₂, Ag₂O, MgO, SnO, TiO₂, B₂O₃, WO₃, As₂O₅, and PbO in place of CuO.

L10 ANSWER 8 OF 9 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1968:49085 CAPLUS

DOCUMENT NUMBER: 68:49085

TITLE: Citric acid

INVENTOR(S): Wiley, Richard H.

SOURCE: U.S., 4 pp.

CODEN: USXXAM

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 3356721	-----	19671205	US	19620321 <--

GI For diagram(s), see printed CA Issue.

AB The preparation of the title compound (I) comprised (a) air oxidation of a 2-butene-1,4-dioic acid over a Pd salt catalyst to give the corresponding oxalacetic acid di-(lower alkyl) ester or anhydride; (b) condensing the ester or anhydride with O:C:CH₂ (II) in the presence of a ZnCl₂-HOAc complex, BF₃, or Al₂O₃-SiO₂-B₂O₃ catalyst to form the β-lactone; (c) hydrolysis of the β-lactone with Ca(OH)₂ to obtain the Ca salt, and subsequent treatment with H₂SO₄. Di-Me fumarate 10 was blown with air 1-5 hrs. in the presence of CuCl₂-Pd catalyst 0.1 part, until a maximum concentration of the di-Me oxalacetate, MeCO₂CH₂COCO₂Me, (III) was obtained. III was identified by spectrometric anal. and the 2,4-dinitrophenylhydrazone, m. 110° (decomposition). The catalyst was separated by filtration and ion exchange treatment, the resulting mixture (containing 40-95% III) treated with 0.5 part ZnCl₂-HOAc complex and II (obtained from diketene) in an amount equivalent to the III present, kept 1 hr. to complete reaction, and the resulting intermediate (β-lactone), 4-carbomethoxy-4-carbomethoxymethyl-2-oxetanone (IV) hydrolyzed with Ca(OH)₂ to give Ca citrate, which on acidification with H₂SO₄ yielded I. IV might be isolated prior to hydrolysis by distillation in vacuo; IV heated to 40-60° gave a polymeric product, which with heat yielded the di-Me aconitate and, and itaconate resp. An Et₂O solution of the Et analog of III (V) containing 16 g. ester was obtained by the Et₂O extraction of an aqueous solution of Na oxalacetate, neutralized with dilute H₂SO₄, and the mixture treated with BF₃-etherate (0.2-0.5 weight % fluoride), then with 4.2 g. II at 0-10°, the mixture kept an hr., and the Et₂O evaporated at low temperature to give the Et analog (VI) of IV. VI on controlled reaction with an equivalent amount cold H₂O was converted to asym. di-Et citrate (VII) (Ag salt m. 110°) which was isolated as a syrup. VII was converted to its Ac derivative, to citric acid, or to tri-Me citrate. Alcoholysis (EtOH) of VII gave tri-Et O-ethylcitrate. VI heated lost CO₂ to give di-Et itaconate on fractionation. Heating the crude oxetanone with a trace of Bz₂O₂ initiator gave a foamed polymer of di-Et itaconate. Hydroxymaleic anhydride (VIII) analogously treated gave the β-lactone intermediate, 1,6-dioxaspiro[3.4]octane-2,5,7-trione (IX). IX warmed gently to apprx.25° with H₂O yielded I. VIII, m. 81-2° 11 (prepared by reaction of diacetyl tartaric anhydride with C₅H₅N at -5° was dissolved in Et₂O 80 parts treated with an equivalent amount of

II in the presence of BF₃ (1.0 weight %) at 0-5° gave IX, which was recrystd. at low temperature. A stirred solution of 17 g. V in 10 ml. Et₂O over 0.5 g. Al₂O₃-SiO₂-B₂O₃ catalyst (U.S. 2,462,357) was treated with 4.2 g. II at 20-5° to give pure IV, as a semisolid which hydrolyzed to I. IX was similarly obtained to give I, m. 98-100°. Air was passed through a suspension of 0.2 g. fumaric acid in 5 ml. concentrated HCl containing 0.1 g. PdCl₂, during 15 min. An aliquot portion of the solution gave the 2,4-dinitrophenyl-hydrozone of oxalacetic acid, m. 170°. Decationization of the reaction mixture on an ion exchange resin followed by evaporation gave pure oxalacetic acid, m. 150.2° (EtOAc). A solution of 10 g. maleic anhydride in 1 l. C₆H₆ was allowed to flow downward over 200 ml. supported Pd catalyst in the presence of a rising stream of air during 4 hrs. The catalyst was prepared by converting a cation exchange resin (Dowex 50-X8, a sulfonated styrene) to the Pd salt by exchange with a PdCl₂ solution or depositing PdCl₂ or PdSO₄ over a **metal oxide** on alumina-silica, alumina-zirconia, or alumina-titania. The effluent C₆H₆ was treated with 0.5 ml. BF₃ etherate and 4.2 g. II to give IX. The depalladinized solution was distilled in vacuo to sep. the solvent, the reactant, and V.

L10 ANSWER 9 OF 9 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1910:5058 CAPLUS

DOCUMENT NUMBER: 4:5058

ORIGINAL REFERENCE NO.: 4:867i, 868a-i, 869a

TITLE: Latest Methods of Manufacturing Metallic Lamp Filaments

AUTHOR(S): Duschnitz, B.

SOURCE: Elec. Rev. and West. Elect. (1910), 56, 32-33, 89-91

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

AB An abstract and r.acte.esum.acte.e of foreign patents on the subject during the past year. Processes are divided into three divisions: (1) "With the aid of organic substances." (2) "From substances free from C by the wet process." (3) "From metallic masses free from C without the use of fluids." (2) is subdivided into (a) "with colloids" and (b) "from combinations of fluid substances." (1) The Wolfram Lamp Co., of Augsburg, Germany, patent a filament of a mixture of W and a small quantity of more easily fusible metals for sintering it, as Cr, V, Nb, Ta, Os, Ru, Zr, or Th. They claim greater strength and elasticity. W powder with 15% or more of above metals is mixed with a ZnCl₂ or acetic acid solution of cellulose, formed into filaments, carbonized and the C removed by one of the regular processes. Siemens and Halske patent the removal of C from filament by heating in an atmos. of acetic acid at reduced pressure. The decarbonization is said to be very rapid and thorough. E. Goosens Pope & Co., Holland, mix a Na tungstate solution with not more than 8% gallic acid and precipitate with acid. The precipitate is a mixture of low W oxides and pyrogallol. It is washed with water and 96% alc. The black shiny paste obtained is formed into filaments, dried slowly, heated to 600° in CO₂ leaving the filament a mixture of W **metal, oxide** and carbide, then in H₂ to reduce oxide and decompose carbide. The French Auer Co., winds the filament pressed from a mixture of W and organic binder on the mount, fires in special furnace in H₂ and completes sintering by passing current through the filament. The mount is then placed in N and filaments glowed to drive H from metallic parts of mount. If this is not done the occluded H escapes slowly during burning and gives a bad vacuum. The Zirkon Lamp Co. remove carbon by means of N or nitrogenous gases produced in vacuum from H-and N-containing P comps., as phospham, phosphamides and phosphamine or their sulphates. Phospham (PN₂H) is especially suitable, decomposing at low temperature and liberating N and P. The process is carried out by (1) mixing the phospham with the metal and binder before pressing, or (2) dipping the filaments in an alc. suspension of phospham before sealing in or (3) dusting the filaments with phospham. Decarbonization may be done during exhaustion. (2a) Johann Lux, of Vienna, presses a paste made of colloidal tungstic acid and W powder. After drying, current is passed through the filaments in an atmos. of H to reduce acid to W. Hans Kuzel finds that the addition to his regular colloid of 5-20% of oxide or hydroxides of refractory metals increases the cohesion of the wet

as well as the dry material. Hollow spaces are quite frequent in the filaments. To avoid this he glows them in an inert or reducing atmosphere at a pressure of not more than 150 mm. Hg. The Siemens and Halske Co., find that plastic acid NH₄ tungstate becomes conducting and insol. in H₂O if heated below red heat in an atms. free from O. They now prepare the plastic mass by evaporating NH₄ tungstate solution, acid NH₄ tungstate separating out. This is filtered off and heated gradually to 120-150°, then to 270° in vacuum (20 mm.Hg). Resulting powder is put in hot H₂O and evaporated to dryness. Residue is a glassy mass. This can be dissolved in hot H₂O and pressed into filaments. J. Lux, Vienna, prepares a binder from NH₄ metatungstate, mixes it with W metal and glows in a reducing atmosphere. (3) Siemens and Halske patent the drawing of Zr and Th or mixtures of these with other metals in a ductile tube of another metal, removing the envelope after drawing by chemical or mechanical means or fusion. They use a spongy mass of Th, etc., obtained by making alloys, and dissolving out the alloying metal. Filaments from a spongy mass are said to be more easily workable than those from powder. The same firm patents the use of powdered W in connection with low evaporating metals as Ag, Ni, Au and Pt. These metals coat the W and the coated W may be pressed into rods and drawn into wire. The low evaporating metal is removed by passing a current. The W powder may be pressed into a tube of Ag, Ni, etc., and the whole encased in an outer tube of steel. This is drawn, the outer tube removed by solvents, the filaments shaped and the low evaporating metal removed by passing a current through the filaments. They also patent a method of making small filaments by heating large filaments in a mixture of P vapor and H at reduced pressure, the cross section being uniformly reduced. The All. Eleckt. Ges. mix W with low m. p. metal as Cu, Cd, Bi, etc., press in a mold, draw, and remove low m. p. metals by a passing current.